SCHOOL OF
CIVIL ENGINEERING

INDIANA
DEPARTMENT OF HIGHWAYS

JOINT HIGHWAY RESEARCH PROJECT

JHRP 86/10

EVALUATION OF AGGREGATE SAMPLE DURABILITY

Margaret A. Hanson

PURDUE UNIVERSITY

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Final Report

EVALUATION OF AGGREGATE SAMPLE DURABILITY

TO: H. L. Michael, Director
Joint Highway Research Project

FROM: W. L. Dolch
Professor

August 27, 1986

Project C-36-470

File 4-6-15

Attached is the Final Report on the JHRP Study entitled "Aggregate Sample Testing for Frost Durability." The title of the report is "Evaluation of Aggregate Sample Durability." The research was conducted by Margaret A. Hanson, Graduate Assistant in Research, under the direction of Professor W. L. Dolch.

The focus of this research was the development of a practical procedure for analyzing the durability of an aggregate source for use in concrete pavement. The recommended method for aggregate sample evaluation, devised to be efficient in terms of time, labor, and expense, is provided.

This report is forwarded for review, comment, and acceptance by the IDOH as fulfillment of the objectives of the study.

Respectfully submitted,

W. L. Dolch

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WLC/mlc

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Final Report

EVALUATION OF AGGREGATE SAMPLE DURABILITY

by

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Project No.: C-36-470

File No.: 4-6-15

Prepared as Part of an Investigation

Conducted by

Joint Highway Research Project
Engineering Experiment Station
Purdue University

in Cooperation with the

Indiana Department of Highways

The contents of this report reflect the views of the author who is responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of the Indiana Department of Hiuhways. This report does not constitute a standard, specification, or regulation.

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August 27, 1986

ACKNOWLEDGEMENTS

The author wishes to thank Dr. Dolch for his wisdom and Dr. Winslow for his ideas. In addition, sincere thanks is given to the other members of her thesis advisory committee, Dr. Virgil Anderson and Dr. Terry West. The financial support of the Indiana Department of Highways was greatly appreciated. A special thanks is due to Janet Lovell for her practical experience. The author also extends much appreciation to Agnes Meyers, Jennifer Landsly, and Patti Fotianos for their computer skill.

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ABSTRACT

Hanson, Margaret A. M.S., Purdue University, August, 1986. Evaluation of Aggregate Sample Durability. Major Professor: Dr. W.L. Dolch.

A method was developed to predict the freeze-thaw durability of an aggregate sample using mercury porosimetry. The foundation of the procedure lies in a statistical subsampling plan to find the minimum number of porosimeter runs necessary to predict the average Expected Durability Factor of a sample, with a predetermined accuracy and precision.

A dolomite and a gravel sample were analyzed in this study. First, a sample was subdivided according to lithologic and textural differences. Next, the absorption of each fraction was determined. The purpose of subdividing and testing the absorption was to decrease the overall amount of analysis time. Crushing the rock samples was also investigated, however a practical method was not found. Those fractions with a high absorption were tested in the mercury porosimeter.

The EDF was determined from the pore size distribution of a sample. Then, the minimum number of EDF values was calculated, based on the variance of the data and on the confidence conditions. Six combinations of confidence coefficients and intervals were compilied. Based on the results of the comparison, a recommended method for aggregate source evaluation was formulated. The test was constructed to be efficient in terms of time, labor and expense.

INTRODUCTION

D-cracking is a major source of distress to Indiana concrete highways. Cyclic freezing and thawing causes the breakdown of nondurable coarse aggregate and of the cement paste surrounding the aggregate. Cracks, running parallel to the joints and edges of slabs, are characteristic of this difficulty (1). As the deterioration advances, the integrity of the entire pavement is destroyed.

Problem

Many methods have been developed to predict aggregate durability in concrete, however none these tests are completely satisfactory (2). Tests that cyclically freeze and thaw concrete containing the aggregate are reasonably successful, although time consuming (3,4). A test that evaluates the rock alone is more desirable because less time, expense and labor are involved. Two such methods are the absorption and sulfate soundness, however, these tests have only a limited success (5,6).

Pore size distribution analysis, using mercury porosimetry, is a recently developed method to predict the freeze-thaw durability of coarse aggregate for concrete

(7,8,9). The method defines an expected durability factor, EDF, that is derived from the pore size distribution curve of a sample. A low EDF indicates a comparatively non-durable aggregate. The borderline between a durable and nondurable EDF has been established, and the maximum allowable percentage of nondurable aggregate in a pavement is also known (10,11). However, the present state of the research is not directly applicable to predict the durability of an aggregate source, because a reliable testing program has not been deveolped.

Objective

The objective of this research is to develop a testing program to evaluate the suitability of a coarse aggregate source for use in concrete pavement. The foundation of the procedure lies in a subsampling statistical analysis to find the minimum number of pore size distributions needed to predict the representative average EDF of a sample with a predetermined degree of accuracy and precision. With such a scheme, a potential source of aggregate could be evaluated in terms of its expected durability in concrete. The ultimate goal of this research is to develop a manual for the application of the EDF method. The method will allow a sample to be evaluated with an efficient use of time, labor, and expense.

Approach

The approach used in this research began with a preliminary investigation of a crushed limestone sample. The purpose of this step was to determine if statistics could be used to analyze the EDF data. A scheme of analysis using lithologic and textural separation, absorption testing, and crushing was proposed.

The next step was to apply the method to a gravel sample. The process of subdividing the gravel required a small amount of revision and the initial scheme was also revised. After the statistical results of all of the EDF data were evaluated, a recommended method for aggregate source evaluation was devised.

Literature Review

Aggregate durability has been studied for many years in an effort to find a solution to D-cracking. In 1924, Reagel observed popouts caused by nondurable chert particles (12). Unsound coarse aggregates were characterized by a low bulk specific gravity, a high absorption, and a high degree of saturation, by 1940 (13).

In 1960, Verbeck and Landgren analyzed the behavior of a rock when it freezes (14). They postulated three types of failure mechanisms for concrete aggregates. The first theory states that frozen water can be accommodated within a rock by elastic expansion. Failure occurs if the aggregate is critically saturated and the volume of frozen water is greater than the expansion potential of the rock. The second mechanism holds that the excess volume of water generated by freezing will be forced to flow through the aggregate. If the hydraulic pressure generated by this movement is too great, the rock will fail internally. A popout is an example of this type of failure. The third failure mechanism occurs when the porosity of the rock is too great for elastic expansion and the permeability is so large that the critical freezing distance is greater than the length of the rock. The excess water generated during freezing is driven into the paste, freezes, and causes the paste to fail. This mechanism is termed expulsion into the paste.

The importance of the pore characteristics of a rock were described by Rhoades and Mielenz in 1946 (15). The volume, size, and continuity of the void structure of an aggregate affects its durability . Dolch concluded that the rate of increase of the degree of saturation and the ratio of the absorptivity to the permeability are two important indicies of frost susceptibility (16).

Dolar-Montuani stressed that the composition affects the pore properties of a rock (17). Sedimentary and partially metamorphosed rocks are frequently responsible for D-cracking. Common nondurable coarse aggregates range from pure carbonates to carbonates containing chert and clay impurties to pure chert, shale, sandstone, greywacke, and metagreywacke. These nondurable aggregates are characterized by a large number of small pores.

In 1921, Washburn first proposed mercury intrusion to measure the distribution of voids in a porous material (18). The first application of porosimetry to correlate the pore characteristics of a rock with pavement performance was conducted by Lemish, Rush, and Hiltrop (19). Conflicting reports about what pore size range characterized nondurable rocks were published in the 1960′s. However, reports agreed that aggregates with a large total pore volume or a large number of pores smaller than 0.1 microns caused distress in concrete pavements (20).

Kaneuji, Winslow, and Dolch developed a correlation between the pore size distribution and the freeze-thaw durability of coarse aggregate in concrete (21). Carbonate, shale, sandstone, and brick samples were selected to represent a wide range of pore sizes. The aggregates were subjected to four durability tests; the rapid freeze-thaw, the modified critical dilation, the vacuum saturated 24-hour absorption, and the Portland Cement Association absorption-adsorption.

The rapid freeze-thaw method gave the best match with the pore size distributions of the samples. Therefore, a correlation equation between the two parameters was obtained by multiple regression analysis. A numerical value of the expected durability was obtained with the following equation:

$$EDF = \frac{0.579}{PV} + 6.12(MD) + 3.04$$

where:

EDF = Expected Durability Factor

PV = Intruded volume of pores larger than 45 Å, cc/g

MD = Median diameter of pores larger than 45 Å, μm

A lower limit of 45 Å was set on the pore diameter because water does not freeze in pores smaller than 45 Å at typical outdoor temperatures (22).

Kaneuji also proposed a borderline between durable and nondurable EDF values. This number was obtained by examining concrete pavements exhibiting D-cracking. The physical description of representative pieces of aggregate was compared with the EDF of those pieces. Rocks from distressed concrete had lower EDF values. The result of the analysis established the following regions: below 40, nondurable aggregate; between 40 and 50, marginal; above 50, durable aggregate.

Another result of Kaneuji´s research suggested a new failure mechanism for freezing aggregate. Water in the smallest pores of a rock freezes at a lower temperature than water in the larger pores of cement paste. Consequently, an expanding aggregate may be confined by the previously frozen cement paste. A dilation type of failure mechanism occurs, because the rock can not expel the excess water created by freezing.

Winslow, Lindgren and Dolch determined what percent of nondurable coarse aggregate in concrete is detrimental to pavement durability (23). This research also refined Kaneuji´s EDF dividing line between durable and nondurable rocks. Indiana highways exhibiting D-cracking were evaluated and sampled. The Portland Cement Association pavement condition rating was used to judge the severity of D-cracking. The amount of distress was judged, by several persons, on a scale of 0 to 5. A rating of 0 means that no damage was visible, and a score of 5 means extensive D-cracking was evident. The average EDF of the significant part of the aggregate was determined by calculating separate EDF values for the durable and nondurable portions. Lindgren determined the relative percentage of each aggregate constituent microscopically, by the point count method (24).

The data were analyzed to construct correlations between the performance rating, the EDF of a given portion of the aggregate, and its volumetric proportion of the total aggregate. The research indicated that an EDF of over 50 represents a durable aggregate. It was further concluded that more than 10% nondurable coarse aggregate in a pavement will result in D-cracking. Therefore, 90% of the aggregate must have an EDF of above 50 to ensure durable concrete.

EXPERIMENTAL WORK

Materials

Two aggregate sources were evaluated in this study. The samples were assumed to be representative of their sources. A crushed dolomite was tested first. This sample, number 106, was obtained from the Indiana Department of Highways and was part of a joint Indiana and Illinois durability testing program. The gravel sample was obtained from a West Lafayette, Indiana terrace deposit.

Equipment

The mercury porosimeter used in this study was manufactured by the American Instrument Company, catalog number J5-7125D. The theory behind porosimetry has been extensively reviewed (25). The usual experimental techniques and data analysis were used in this research. A surface tension value of 484 dynes/cm and a contact angle of 125 degrees were assumed, based on the work of Lindgren (26). The pressure generated by the porosimeter ranges from less than 0.01 mm of mercury to a maximum of 60,000 psi. The corresponding range in measured pore diameter is from about 500 microns to about 25 Angstroms.

The porosimeter was previously modified to suit the specialized needs of rock measurements. A fan was installed to decrease the amount heat generated by compressing the hydraulic fluid. Also, two low pressure measuring devices were added to allow pores in the 10 to 500 micron range to be determined more accurately. The pressure vessel cap was modified to accomodate a large sample holder.

Two sizes of sample holding devices, or penetrometers, were used. The smaller penetrometer had a 0.2 ml stem, or intrusion capacity and a bulb that held approximately five grams of sample. The larger penetrometer had a 2.0 ml intrusion capacity and held a sample of about 20 grams.

Corrections

Four correction factors were needed to offset the error in the measured intrusion. The first correction is necessary because a perfect vacuum is not achieved in the penetrometer. The extra gas occupies a volume, which decreases as the pressure increases. Therefore, the apparent intrusion is greater than the true intrusion, and a correction factor must be subtracted from the measured value. At pressures of less than one atmosphere, this correction is significant. Above one atmosphere the decrease in the gas volume is constant, but the correction must still be subtracted from the apparent intrusion. The value of the factor is determined from Boyle´s law.

The error in the high pressure regime is a combination of three phenomena. As pressure increases, the volume of the sample decreases, leading to apparent intrusion. The penetrometer is also compressed at high pressure, but this change causes extrusion of mercury. The third effect of increasing pressure is a rise in the temperature of the mercury. The last factor is important because the density of mercury is sensitive to temperatue. As the temperature increases the density of mercury decreases, leading to apparent extrusion. The net high pressure error, the sum of these three phenomena, is extrusion. The high pressure correction factors are given in Table 1.

TABLE 1

HIGH PRESSURE CORRECTIONS

PRESSURE (psi)	CORRECTION (ml/ml of Hg in penetrometer)
30	0.00000
50	0.00000
90	0.00002
150	0.00003
250	0.00005
450	0.00008
800	0.00011
1,300	0.00015
2,300	0.00020
4,000	0.00026
7,000	0.00034
12,000	0.00042
20,000	0.00051
35,000	0.00063
60,000	0.00076

Procedure

The first step in the evaluation of the freeze-thaw durability of an aggregate source was to separate the samples into groups of similar composition and texture. The second step was to determine the absorption of each class. Then, those fractions with a comparatively high absorption were tested in the porosimeter. The applicability of crushing aggregates was also examined.

Subdivision

The aggregates were separated according to lithologic and textural characteristics that were expected to lead to differences in porosity. Subdividing reduces the variability of a given sample, therefore, the amount of porosimeter testing was expected to decrease. On the other hand, a division into many fractions that have a very low variability, will require a large amount of testing. A balance was desired between the labor of separation and the labor of testing needed to predict the EDF.

Basic geologic tests were used to separate the rock samples. Color, grain size, cleavage, hardness, and acid reaction give important information about the type and condition of the aggregates. A combination of these characteristics is diagostic for a specific rock type (27).

After washing, the rocks were visually examined. Scratch hardness was determined with a steel file and a penny. A solution of ten percent hydrochloric acid was used to identify limestones. A hand lens was also used to examine fine-grained rocks.

Dolomite. The dolomite sample was relatively homogeneous, even though variations in color and porosity were evident. The overall physical condition of the sample was good. The rocks ranged in size from 1/2 inch to 1 1/2 inches. The dolomite was fine-grained, light to dark gray in color, slightly to very porous, and contained some clay seams.

The dolomite was subdivided into five fractions, based on variations in the general characteristics. Fraction 1 was light gray, with a small amount of pores and clay seams. The second class was similar to the first, but darker gray in color. Fraction 3 contained a high proportion of clay seams. Fraction 4 had a relatively large amount of vuggs, ranging in size from one to three millimeters. The fifth fraction was a red brown color.

Gravel. The overall condition of the gravel was good. The pieces ranged in size from 1/4 inch to two inches; most of the rocks were one inch in diameter. The gravel was composed of angular and well-rounded aggregates.

Separating the gravel was complicated, because the sample was heterogeneous. The division scheme was revised during four trials to find the best classification scheme. The weight percentages of each gravel subdivision was compared to that of a typical Indiana gravel (28). The values matched closely, therefore, the accuracy of the subdivision procedure was reinforced. Igneous and metamorphic rock had similar characteristics, so they were grouped together. The shales and ochers had different characteristics compared to the other terrigenous sedimentary rocks, so they were put into separate classes. Chert and quartzite have different pore structures, therefore, chert was put in a separate catagory and quartz was placed with the igneous and metamorphic rocks. Quartzites were classified with the sandstones. The division between weathered and fresh rocks was also more clearly defined in the course of the four trials. Table 2 lists the distinguishing characteristics for each rock type.

TABLE 2

DISTINGUISHING CHARACTERISTICS OF GRAVEL SUBDIVISIONS

ROCK TYPE	COLOR	HARDNESS	GRAIN SIZE	OTHER FEATURES
Igneous and Metamorphic	clear, white, pink, brown, black	6 hard	fine to coarse	cleavage, metamorphic fabric
Chert	cream, tan	7 very hard	microcrystalline	conchoidal fracture
Sandstone	brown, tan	3-6 variable	medium to coarse	friable
Ocher and Shale	brown, black red	2 very soft	very fine	bedding
Limestone	gray, tan	3 soft	fine	efferveses with acid, may be fossiliferous
Dolomite	tan, brown	3 soft	fine	no effervesence with acid, may be fossiliferous

The final scheme was to divide the gravel into eleven catagories:

1. Igneous and Metamorphic
2. Weathered Igneous and Metamorphic
3. Chert
4. Weathered Chert
5. Ocher and Shale
6. Sandstone
7. Weathered Sandstone
8. Limestone
9. Weathered Limestone
10. Dolomite
11. Weathered Limestone

The following general procedure was used to subdivide the gravel:

1. Sort out obvious rocks.
2. Perform acid test on remaining rocks.
3. Perform hardness test on remaining rocks.
4. Sort out weathered rocks.

The first step was visually to separate all of the rocks that were easy to identify. The recognition of some types of igneous rocks was obvious: granite, diorite, and vein quartz. The microcrystalline grain size of chert was characteristic. Some of the sandstones and limestones were also easy to identify.

The next step was to test the remaining unidentified pieces with a drop of acid. Any rock that contained a high proportion of limestone effervesed profusely in acid. The physical appearance of the limestone was variable, so testing each rock was important. Some of the rocks effervesed only slightly. Limestones with clay or chert impurities were separated according to the relative amount of the impurity.

The remaining rocks were classified by the hardness test. Each piece was scratched with a steel file. Hard rocks exhibiting cleavage, relict grains, or a wavy metamorphic texture were placed in the igneous and metamorphic class. Cherts were extremely hard, microcrystalline, and were characterized by conchoidal fracture. Sandstones were identified by a coarse grained texture and typically friable nature. The shale and ocher fraction was soft enough to be scratched deeply. Dolomite rocks were distinguished by a moderate hardness and a brown to yellow-brown to tan color.

Problems occured when rocks could not be identified precisely. The distinguishing characteristics of deeply weathered rocks and of fine grained dark rocks were difficult to determine. In these cases, the rock was placed in the class that matched most of the characteristics. The dark, fine-grained sandstones and some of the andesites had almost identical physical properties. Both of the rocks had similiar pore characteristics, therefore, the rock was acceptable in either catagory.

The fourth and final step of the gravel subdivision procedure was to separate the weathered and the unweathered rocks within one lithologic fraction. This step was to some extent, subjective because the degree of weathering graded from slightly to deeply weathered. Separating the gravel was complicated because the surface of a piece of gravel was more weathered than the core. A rock that appeared weathered on the outside, was frequently fresh and durable on the inside. The distinguishing features of a highly weathered rock were a dull and earthy color, a high proportion of relict grains converted to clay minerals, a low relative hardness, and a low relative specific gravity. As a rule, only deeply weathered rocks had a large change in pore properties, and only a small proportion of each fraction was highly altered.

Absorption

The second step in the experimental procedure was to determine the absorption of each aggregate fraction. The ASTM recommended method for absorption, C-127, was used. The purpose of this step was to screen out groups with an absorption of less than one percent. The EDF of such materials will always be greater than about 60, therefore, those rocks will always be durable. Eliminating these subdivisions significantly reduced the amount of testing.

Porosimeter

Testing the rock fractions that had a high absorption in the mercury porosimeter was the next step in the experimental procedure. A large set of data was needed to establish the statistical procedure. Fifteen runs, on both the light and dark gray dolomite subsamples, were performed. The other three classes were each tested eight times. The intrusion was measured at pressure multiples of approximately 1.75, which allowed the entire pressure range to be covered uniformly and graphed easily. The high pressure intervals are given in Table 1. The low pressure intervals were 20, 40, 80, 160, 320 and 640 mm of mercury. The small penetrometers were used to test the crushed stone, therefore the weight of each sample was approximately five grams. The individual test samples were chosen randomly.

The procedure for testing the gravel in the porosimeter was slightly different. Large penetrometers were used, therefore, the sample size was increased to between ten and twenty-five grams. The weight of the sample was dependent on the absorption of the rock. The low pressure intrusion intervals were also changed. The sample was intruded initially to 200 mm Hg and measured at 320 and 640 mm Hg. A large initial intrusion offset part of the interparticle pore volume error. The rocks for each porosimeter sample were chosen to represent the range of textures present in the whole fraction. This method was viewed as another type of subdivision. Three runs were performed on a class, the statistics were calculated, then four more runs were made and the statistics were recalculated based on seven runs.

Crushing

Using a sample of crushed material was investigated as another method to reduce the amount of testing needed to predict the EDF of an aggregate fraction. A sample containing many small pieces has a wide variety of the textures found in the class. The composite pore size distribution of many rocks makes the measured EDF of the sample closer to the true value. The variability among samples is also less, so the amount of testing required decreases. The practicallity of this idea was investigated for both the dolomite and the gravel sources.

The dark gray class of dolomite was crushed with a hammer to pass the number four sieve and be retained on the number eight. The sample was washed on the sieve and oven dried prior to testing. Normal intrusion intervals were used. Eleven runs were performed on the dolomite.

The problem with the initial scheme was that the crushed sample contained a comparatively large amount of space between the rock grains that was recorded by the porosimeter along with the true rock pores. So, the pore size distribution was biased towards an artificially high amount of pores on the large diameter end of the distribution.

Tests were done to try to refine the crushing procedure. Two methods were investigated on the unweathered dolomite fraction of the gravel to find an effective way to correct the large intergranular pore volume created by crushing.

In the first method, quartzite was used as a standard. Quartzite has virtually no pores, therefore the only measured intrusion must be due to interparticle pore volume. Samples with a particle size range from 3/8 to 1/4 inch were tested. Five runs were performed on the quartzite and ten were run on the dolomite.

The aim of the other correction method was to intrude mercury beyond a point where any intergranular pore volume remained. Theoretically, the pore size distribution graph should level out at the point where all of the interparticle pore volume has been intruded. Crushed dolomite, ranging in size from 3/8 to 1/4 inch, were intruded at normal intervals.

DATA

Porosimeter

The sample weight, vacuum reading, cummulative intrusion and other important instrument readings were used to determine the pore size distribution of a sample. A special form was used to record this information, however the raw data will not be included in this report because of the large volume involved. Over 125 porosimeter runs were conducted during the course of the study.

RESULTS

Absorption and Weight Percent

The weight percent of each rock catagory was calculated from the oven dry weight. The percent absorption of a fraction was calculated from the oven dry and saturated surface dry weights of each fraction according to ASTM C-127. Table 3 lists the weight percents and the percent absorptions for the dolomite; the results for the gravel are given in Tables 4 through 7.

Porosimeter

The EDF was calculated with the use of a computer program that incorporated the correction factors into the measured pore size distribution. The computer printouts were not included in this report for the same reasons that the raw data sheets were not given. Table 8 gives the EDF values and averages for the dolomite. The gravel EDF values, after three and six runs, are listed in Tables 9 and 10, respectively.

TABLE 3

DOLOMITE WEIGHT AND ABSORPTION PERCENTAGES

TYPE NO.	ROCK TYPE	WEIGHT PERCENT	PERCENT ABSORPTION
1	Light Gray	23.8	1.7
2	Dark Gray	57.8	2.1
3	Clay Seams	5.2	2.6
4	Porous	6.3	1.8
5	Red-Brown	6.8	2.3

TABLE 4

GRAVEL NO. 1 WEIGHT AND ABSORPTION PERCENTAGES

TYPE NO.	ROCK TYPE	WEIGHT PERCENT	PERCENT ABSORPTION
1	Igneous and Metamorphic	17.5	0.3
2	Weathered Igneous and Metamorphic	12.9	1.1
3	Quartzite and Chert	3.4	0.5
4	Weathered Quartzite and Chert	2.7	2.4
5	Sandstone	8.5	0.9
6	Weathered Sandstone	6.9	4.0
7	Limestone	15.2	0.8
8	Weathered Limestone	9.6	3.1
9	Dolomite	23.3	1.9

TABLE 5

GRAVEL NO. 2 WEIGHT AND ABSORPTION PERCENTAGES

TYPE NO.	ROCK TYPE	WEIGHT PERCENT	PERCENT ABSORPTION
1	Igneous and Metamorphic	31.4	0.4
2	Weathered Igneous and Metamorphic	6.3	2.3
3	Chert	4.4	1.6
4	Weathered Chert	2.6	3.3
5	Ocher and Shale	3.8	6.8
6	Sandstone	10.1	0.5
7	Weathered Sandstone	2.7	3.5
8	Limestone	14.7	0.7
9	Weathered Limestone	5.9	2.9
10	Dolomite	18.1	1.9

TABLE 6

GRAVEL NO. 3 WEIGHT AND ABSORPTION PERCENTAGES

TYPE NO.	ROCK TYPE	WEIGHT PERCENT	PERCENT ABSORPTION
1	Igneous and Metamorphic	32.6	0.4
2	Weathered Igneous and Metamorphic	7.3	1.5
3	Chert	6.3	1.3
4	Weathered Chert	2.2	3.8
5	Sandstone	5.1	1.1
6	Weathered Sandstone	1.3	5.0
7	Ocher and Shale	1.8	7.9
8	Limestone	13.4	0.7
9	Weathered Limestone	2.7	2.6
10	Dolomite	21.8	1.7
11	Weathered Dolomite	5.5	4.3

TABLE 7

GRAVEL NO. 4 WEIGHT AND ABSORPTION PERCENTAGES

TYPE NO.	ROCK TYPE	WEIGHT PERCENT	PERCENT ABSORPTION
1	Igneous and Metamorphic	34.6	1.0
2	Weathered Igneous and Metamorphic	2.6	2.3
3	Chert	3.8	1.1
4	Weathered Chert	4.7	3.7
5	Sandstone	3.4	0.8
6	Weathered Sandstone	1.2	4.5
7	Ocher and Shale	1.6	9.5
8	Limestone	12.6	0.6
9	Weathered Limestone	3.1	2.1
10	Dolomite	24.4	0.3
11	Weathered Dolomite	8.0	3.5

TABLE 8

DOLOMITE EDF VALUES AND AVERAGES

TYPE NO.	ROCK TYPE	EDF VALUES	AVERAGE EDF
1	Light Gray	32,23,25,38,31,29,30,28, 46,32,27,29,35,27,28	31
2	Dark Gray	34,27,41,30,35,22,48,43, 22,35,29,39,23,40,37	34
3	Clay Seams	25,26,25,28,25,26,23,25	25
4	Porous	23,25,33,31,34,46,30,25	31
5	Red-Brown	42,21,39,22,20,23,26,24	27

TABLE 9

GRAVEL EDF VALUES AND AVERAGES BASED ON THREE RUNS

TYPE NO.	ROCK TYPE	EDF VALUES	AVERAGE EDF
2	Weathered Igneous and Metamorphic	23,31,28	27
3	Chert	22,17,32	24
4	Weathered Chert	13,20,16	16
6	Weathered Sandstone	16,18,14	16
7	Ocher and Shale	14,8,10	11
9	Weathered Limestone	16,24,21	20
11	Weathered Dolomite	24,20,19	21

TABLE 10

GRAVEL EDF VALUES AND AVERAGES BASED ON SEVEN RUNS

TYPE NO.	ROCK TYPE	EDF VALUES	AVERAGE EDF
2	Weathered Igneous and Metamorphic	23,31,28,46,29,40,33	32
3	Chert	22,17,32,25,24,26,29	23
4	Weathered Chert	13,20,16,22,15,20,20	18
6	Weathered Sandstone	16,18,14,14,15,20,14	16
7	Ocher and Shale	14,8,10,9,13,10,8	10
9	Weathered Limestone	16,24,21,22,31,26,28	24
11	Weathered Dolomite	24,20,19,15,17,17,21	19

DISCUSSION

Experimental Procedure

Crushing

The average EDF of the crushed dolomite was significantly lower than the average EDF of the uncrushed dolomite. The average EDF for the crushed samples was 22 and the average EDF for the whole rocks was 34. The averages were expected to be close, and the spread of the crushed rock EDF values was expected to be smaller. The range of the crushed dolomite EDF values was 21 to 23, and the uncrushed samples had a range of 22 to 48.

Pore size distribution graphs of the crushed and the whole pieces of dolomite were compared. The crushed samples had a much larger pore volume in the low pressure range. This extra volume was due to interparticle pore space. The rock grains in a penetrometer are in contact with the sides of the penetromter and also with other grains. The grain boundries act like large pores. When the pressure increases to a point where the rocks no longer touch, the interparticle error is eliminated. The apparent intrusion is greater than the true intrusion, therefore, the EDF of the crushed dolomite samples was too low. This error also occurred in the uncrushed samples, but the number of grains was small, so the interparticle pore volume was small.

The attempt to use crushed samples was more complicated than anticipated. A correction factor was needed to make crushing a more practical method. Two ways were investigated to correct the interparticle pore volume error. The quartzite correction factor was not effective because the surface area of the quartzite was less than the dolomite. The dolomite grains were very rough and created a larger intergranular pore volume than the quartzite. The second approach was to subtract the interparticle pore volume from each run. Unfortunately, the distinction between interparticle and intraparticle pores was not clear, and the spaces between the grains were not fully intruded in the low pressure regime.

These correction attempts with the crushed samples were not successful. Crushing was concluded to be an unpractical method to reduce the amount of testing needed to represent a sample. Therefore, the gravel samples were analyzed using whole pieces.

Absorption

Dolomite. The dolomite class rich in clay seams had the highest absorption value. The red-brown colored dolomite subsample also had a relatively high absorption, clay was probably an important constituent of this rock. The dark gray dolomite had an absorptivity between that of the clay-rich, and the light gray and the vuggy classes.

All of the dolomite fractions had an absorption of greater than one percent; therefore, each of the five subsamples was tested in the porosimeter. When the absorptions of all of the classes were compared, the results were similar. Given the uncertainty of the saturated surface dry condition, the percent absorption of these subdivisions were basically equivalent. Based on this result, the EDF values were predicted to be close.

Gravel. The gravel absorption values had a large variability. The range in the percent absorption between unweathered and weathered classes was also significant. These comparisons are one indication that the subdivision procedure was effective and will reduce the overall amount of time spent analyzing the gravel source.

The weathered fractions of all of the rock types had an absorption greater than one percent. The unweathered chert and the ocher and shale classes also had a high absorption. Therefore, seven subdivisions required further testing in the mercury porosimeter.

Statistics

Review

The statistical background for this study is reviewed in the following section (29). Experimental data are summarized graphically by a frequency distribution. The number of occurrences of each value versus a given class width illustrates valuable information about a population. A frequency distribution is numerically represented by the mean and the standard deviation. The mean, $\overline{X}$, is a measure of the center position or highest number of occurrences in a frequency distribution. The equation for calculating the mean is:

$$\overline{x} = \sum_{i=1}^{n} \frac{x_i}{n}$$

where:

i = index

n = total number of data values

x_i = value of data

The standard deviation, s, and the variance, s^2, measure the dispersion of data. The sum of squares, ss, measures the deviations around the mean. The variance and the standard deviation are given by the following equations:

$$s^2 = \frac{ss}{n-1} = \sum_{i=1}^{n} \frac{(x_i - \bar{x})^2}{n-1}$$

$$s = \sqrt{s^2}$$

A probability distribution represents the likelihood of the occurrence a specific event, based on the frequency distribution. Characteristic distributions are formed; an important one is the normal probability distribution. The normal curve is bell shaped and symmetrical. The mean of a normal distribution is symbolized by μ, and the standard deviation is σ.

Another important probability distribution is the Student-t. When the sample size is small, in the case of the EDF values, data no longer follow a standard normal curve. The t-probability distribution is almost bell shaped and symmetrical. As the size of the sample increases, the t-distribution approches a normal curve.

A probability distribution is numerically expressed by an interval estimate, L. The interval estimate takes into account the error, or precision of the mean, and is represented by the following equation.

$$\bar{x} - L < \mu < \bar{x} + L$$

The value of the interval is determined partially by the standard error, or the deviation of the mean equation.

$$s_{\bar{y}} = \sqrt{\frac{s^2}{n}}$$

The confidence coefficient, α, is the probability that a correct interval estimate is obtained. The coefficient is determined by judging how precise the results must be. The coefficient also affects the width of an interval estimate. The coefficients 66%, 95% and 99% are common values. A 95% confidence coefficient means that in 95% of the occurences, the interval will contain the true population mean.

The confidence coefficient is converted into a percentage point value to calculate the interval estimate. For the t-distribution, the form of the percentage point is:

$$t(1-\frac{\alpha}{2};\ n-1)$$

When α_1 is equal to 99%, the first term in the parenthesis is calculated by the following method.

$$\alpha = 1 - \alpha_1$$

$$.01 = 1 - .99$$

$$1 - \frac{\alpha}{2} = .995$$

$$1 - .995 = .005$$

The degrees of freedom, df, is the other consideration of the percentage point value. Degrees of freedom refers to the number of observations minus the number of parameters estimated from those data values.

$$df = n-1$$

When the sample size is equal to 15, the degrees of freedom is 14. Therefore, if the percentage point terms are t(.005; 14), then $t = 2.977$. The value for t was determined by consulting the table in Appendix A.

The interval estimate, or confidence limits combine the standard error, t-percentage point, and the mean to predict the interval for estimating μ. The limits for the Student-t distribution are given below.

$$\bar{x} - t(1-\frac{\alpha}{2};\ n-1)s_{\bar{y}} \le \mu \le \bar{x} + t(1-\frac{\alpha}{2};\ n-1)s_{\bar{y}}$$

Minimum n

The method used to determine the smallest number of EDF values necessary to predict the range within which the true mean lies, was similar to calculating the confidence limits (30). An interval estimate was broken down into its components:

$$\bar{x} - L \le \mu \le \bar{x} + L$$

$$\bar{x} - t(1-\frac{\alpha}{2};\ n-1)s_{\bar{y}} \le \mu \le \bar{x} + t(1-\frac{\alpha}{2};\ n-1)s_{\bar{y}}$$

$$\bar{x} - t(1-\frac{\alpha}{2};\ n-1)\sqrt{\frac{s^2}{n}} \le \mu \le \bar{x} + t(1-\frac{\alpha}{2};\ n-1)\sqrt{\frac{s^2}{n}}$$

therefore:

$$L = t(1-\frac{\alpha}{2};\ n-1)\sqrt{\frac{s^2}{n}}$$

Two different n values are included in a limit. The n that is contained within the parenthesis is determined by the number of values that the variance was calculated from. The other n, under the square root symbol, is the estimated number of values.

In this study, the confidence limit, confidence coefficient, and the variance were known. The equality of the equation was determined by the estimated value of n. The method can be best explained by a sample calculation. In the next section, the dolomite source was used as an example.

The confidence coefficient and the confidence interval were varied in order to determine the most practical minimum n condition. The purpose of changing these parameters was to create a large data base to compare minimum n values. The effect on the number of runs was expected to be significant. A reasonable number of porosimeter runs, fewer than fifteen, was considered acceptable.

The confidence coefficients used were 80%, 95%, and 99%. These three were chosen to represent the wide range of commonly used confidence coefficients. The confidence interval was tested at L = ± 2 and ± 5. These values were selected based on the certainty of an EDF measurement; an interval of ± 2 is narrower and creates a more precise estimate. The outcome of varying the confidence coefficient and the confidence interval created six conditions, listed in Table 11.

TABLE 11

CONFIDENCE CONDITIONS

CONFIDENCE COEFFICIENT	CONFIDENCE INTERVAL
80%	± 2
80%	± 5
95%	± 2
95%	± 5
99%	± 2
99%	± 5

Dolomite. The classical statistical procedures are based on a normal probability distribution. Before statistical tests can be appplied to the data, the normality must be verified. The W test is a basic statistical method used to establish normality (31). The calculations of the W test were not included in this study, however the results of the tests concluded that the data were normal.

The minimum n statistical method is outlined in detail for the first subsample of the dolomite.

A. Compile data.

28, 32, 23, 35, 38, 31, 29, 30, 28,
46, 32, 27, 29, 35, 27

$n = 15$

B. Calculate mean.

$\bar{y} = 470/15 = 31$

C. Calculate deviation or sum of squares.

$ss = 1 + 64 + 16 + 49 + \ldots + 9 = 431$

D. Calculate variance.

$s^2 = 431/14 = 31$

E. Given confidence condition:

$\alpha_1 = 80\%$ and $L = \pm 2$

1. Estimate value of n required for confidence condition to hold:

$$L = t(1-\frac{\alpha}{2};\ n-1)\sqrt{\frac{s^2}{n}}$$

$n = 20$

a. Calculate percentage point value:

$$t = t(1-\frac{\alpha}{2};\ n-1)$$

$$\alpha = 1 - \alpha_1 = 1 - .8 = .2$$

$$t = 1 - \frac{\alpha}{2} = 1 - \frac{.2}{2} = .9$$

$$1 - .9 = .1$$

$$n - 1 = 19$$

$$t = t(.1;\ 19) = 1.328$$

b. Calculate standard error of estimated n:

$$s_{\bar{y}} = \sqrt{\frac{s^2}{n}} = \frac{31}{20} = 1.245$$

c. Calculate confidence limit:

$$L = t(1-\frac{\alpha}{2};\ n-1)s_{\bar{y}}$$

$$L = (1.328)(1.245)$$

$$L_e = 1.65$$

d. Compare estimated confidence limit with desired confidence limit:

$$L = \pm\ 2$$

$$L_e = 1.65$$

$$L_e < L$$

Conclusion:

The estimated confidence limit is more narrow than required, therefore the estimated n can be decreased.

2. Estimate $n = 15$

a. $t = t(1-\frac{\alpha}{2}; n-1)$

$t = t(.1; 14)$

$t = 1.345$

b. $s_{\bar{y}} = \sqrt{\frac{s^2}{n}}$

$s_{\bar{y}} = \sqrt{\frac{31}{15}}$

c. $L_e = (t)s_{\bar{y}}$

$L_e = (1.345)(2.438)$

$L_e = 1.93$

d. $L_e < L$

therefore, n lower

3. Estimate $n = 14$

a. $t = t(.1; 13)$

$t = 1.350$

b. $s_{\bar{y}} = \sqrt{\frac{31}{14}}$

$s_{\bar{y}} = 1.488$

c. $L_e = (1.350)(1.488)$

$L_e = 2.01$

d. $L_e = L$

TABLE 12

DOLOMITE MINIMUM n RESULTS

TYPE NO.	VARIANCE	ROCK TYPE	EDF ± 2 80%	 95%	 99%	EDF ± 5 80%	 95%	 99%
1	31	Light Gray	14	35	55	4	8	12
2	64	Dark Gray	27	*	*	6	12	21
3	2	Clay Seams	3	5	7	2	3	4
4	52	Porous	23	50	*	5	11	18
5	72	Red-brown	31	*	*	6	13	27
	50	COMBINED SAMPLE	22	50	*	5	11	17

* = Minimum n greater than 60

The result of the calculation determined that fourteen EDF values were needed to properly define the interval within which the true mean lies, when the confidence coefficient is 80% and the EDF confidence limit is ± 2.

The minimum n for the six confidence conditions was calculated for the five dolomite rock types. The results are given in Table 12. Combined sample statistics are also included in Table 12.

The purpose of calculating six confidence conditions was to find the lowest n without sacrificing needed precision. Table 12 shows that the minimum n was senstive to different confidence coefficients and limits. For example, the minimum n almost doubled each time the coefficient was increased. Also, when the limits were expanded from two to five EDF values, the minimum n dropped dramatically. The number of EDF values changed so much because the sum of squares calculation, ss, measures the deviation of data around the mean. As the variability in the data increased, the deviation was numerically squared.

Given the uncertainty of the EDF measurement, a 95% confidence coefficient and an EDF confidence limit of plus or minus five was concluded to be the best confidence condition for the dolomite. This situation created the best balance between accuracy and precision. A five percent chance of not including the true mean in the interval estimate is small. When the coefficient was increased to 99%, the minimum n increased greatly. However, a chance of being wrong 20% of the time was too high. The confidence limit was concluded to be the best best at plus or minus five because the EDF dividing line between durable and not durable is somewhat uncertain. A gray area between 45 and 55 exists where a rock may or may not be durable. The EDF of all of the dolomite subsamples was less than 35.

The combined minimum n conditions, for the crushed dolomite, were determined by grouping the EDF values from the five subsamples together. Although the variances of the fractions were not equal, this situation was realistic because small sample sizes were tested. The variance of the combined sample was less than the variance of three of the subdivisions. This comparison showed that separating the dolomite into classes with similar texture and colors was not necessary. The time spent dividing the sample actually increased the amount of testing required to predict the durability.

The entire dolomite source had an EDF of less than 50. Lindgren's research indicates that an aggregate with greater than 10% non-durable rocks can be expected to show D-cracking in concrete pavements.

Gravel. Tables 13 and 14 give the minimum n results for the gravel, based on three and seven runs, respectively. Comparing the data in the two tables shows that the minimum n did not usually change significantly between three and seven runs. However, the variances of the chert and the igneous and metamorphic classes were very different. When the confidence coefficient was 95% and the confidence limit was plus or minus two EDF values, the resulting minimum n was a reasonable number.

All of the samples tested in the porosimeter had low EDF values. The weight percent of the nondurable gravel classes totaled 25% of the entire sample. A concrete pavement made with this gravel can be expected to exhibit D-cracking, according to Lindgren. However, the extent of the distress will probably not be as severe as in a pavement composed of all nondurable coarse aggregate.

TABLE 13

GRAVEL MINIMUM n RESULTS BASED ON THREE RUNS

TYPE NO.	VARIANCE	ROCK TYPE	EDF ± 2			EDF ± 5		
			80%	95%	99%	80%	95%	99%
2	17	Weathered Igneous and Metamorphic	8	18	30	3	5	8
3	59	Chert	25	61	*	5	12	20
4	13	Weathered Chert	7	14	25	3	5	7
5	9	Ocher and Shale	5	12	19	3	4	6
7	4	Weathered Sandstone	3	6	10	2	3	4
9	31	Weathered Limestone	14	35	55	4	8	12
11	7	Weathered Dolomite	5	9	15	2	4	5

* = Minimum n greater than 60.

TABLE 14

GRAVEL MINIMUM n RESULTS BASED ON SEVEN RUNS

TYPE NO.	VARIANCE	ROCK TYPE	EDF ± 2 80%	EDF ± 2 95%	EDF ± 2 99%	EDF ± 5 80%	EDF ± 5 95%	EDF ± 5 99%
2	67	Weathered Igneous and Metamorphic	30	60	*	6	13	22
3	23	Chert	11	24	40	3	6	10
4	11	Weathered Chert	6	13	22	3	4	7
5	6	Ocher and Shale	4	8	13	2	4	7
7	6	Weathered Sandstone	4	8	13	2	4	5
9	24	Weathered Limestone	11	25	41	3	6	10
11	9	Weathered Dolomite	5	11	19	3	4	6

* = Minimum n greater than 60.

CONCLUSION

A statistical procedure was developed to determine the smallest number of mercury porosimeter runs needed to represent the average EDF of an aggregate source. The minimum n was dependent on the variance, the confidence coefficient, and the confidence interval of a given class of rock. The method was designed to balance the amount of labor spent separating a sample with the labor of testing a sample. A practical procedure was developed to analyze the durability of an aggregate source. The recommended method for aggregate sample evaluation is given in Appendix B.

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REFERENCES

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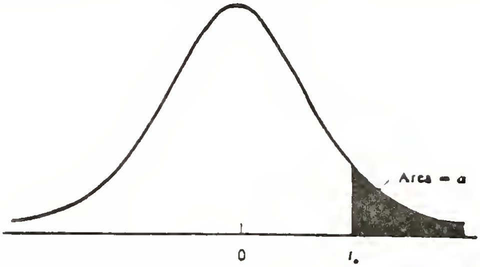
APPENDICES

Appendix A

TABLE 15

PERCENTILES OF THE STUDENT-t DISTRIBUTION



The following table provides the values of t_α that correspond to a given upper-tail area α and a specified number of degrees of freedom.

Degrees of Freedom	Upper-Tail Area α									
	.4	.25	.1	.05	.025	.01	.005	.0025	.001	.0005
1	.325	1.000	3.078	6.314	12.706	31.821	63.657	127.32	318.31	636.62
2	.289	.816	1.886	2.920	4.303	6.965	9.925	14.089	22.327	31.598
3	.277	.765	1.638	2.353	3.182	4.541	5.841	7.453	10.214	12.924
4	.271	.741	1.533	2.132	2.776	3.747	4.604	5.598	7.173	8.610
5	.267	.727	1.476	2.015	2.571	3.365	4.032	4.773	5.893	6.869
6	.265	.718	1.440	1.943	2.447	3.143	3.707	4.317	5.208	5.959
7	.263	.711	1.415	1.895	2.365	2.998	3.499	4.029	4.785	5.408
8	.262	.706	1.397	1.860	2.306	2.896	3.355	3.833	4.501	5.041
9	.261	.703	1.383	1.833	2.262	2.821	3.250	3.690	4.297	4.781
10	.260	.700	1.372	1.812	2.228	2.764	3.169	3.581	4.144	4.587
11	.260	.697	1.363	1.796	2.201	2.718	3.106	3.497	4.025	4.437
12	.259	.695	1.356	1.782	2.179	2.681	3.055	3.428	3.930	4.318
13	.259	.694	1.350	1.771	2.160	2.650	3.012	3.372	3.852	4.221
14	.258	.692	1.345	1.761	2.145	2.624	2.977	3.326	3.787	4.140
15	.258	.691	1.341	1.753	2.131	2.602	2.947	3.286	3.733	4.073
16	.258	.690	1.337	1.746	2.120	2.583	2.921	3.252	3.686	4.015
17	.257	.689	1.333	1.740	2.110	2.567	2.898	3.222	3.646	3.965
18	.257	.688	1.330	1.734	2.101	2.552	2.878	3.197	3.610	3.922
19	.257	.688	1.328	1.729	2.093	2.539	2.861	3.174	3.579	3.883
20	.257	.687	1.325	1.725	2.086	2.528	2.845	3.153	3.552	3.850
21	.257	.686	1.323	1.721	2.080	2.518	2.831	3.135	3.527	3.819
22	.256	.686	1.321	1.717	2.074	2.508	2.819	3.119	3.505	3.792
23	.256	.685	1.319	1.714	2.069	2.500	2.807	3.104	3.485	3.767
24	.256	.685	1.318	1.711	2.064	2.492	2.797	3.091	3.467	3.745
25	.256	.684	1.316	1.708	2.060	2.485	2.787	3.078	3.450	3.725
26	.256	.684	1.315	1.706	2.056	2.479	2.779	3.067	3.435	3.707
27	.256	.684	1.314	1.703	2.052	2.473	2.771	3.057	3.421	3.690
28	.256	.683	1.313	1.701	2.048	2.467	2.763	3.047	3.408	3.674
29	.256	.683	1.311	1.699	2.045	2.462	2.756	3.038	3.396	3.659
30	.256	.683	1.310	1.697	2.042	2.457	2.750	3.030	3.385	3.646
40	.255	.681	1.303	1.684	2.021	2.423	2.704	2.971	3.307	3.551
60	.254	.679	1.296	1.671	2.000	2.390	2.660	2.915	3.232	3.460
120	.254	.677	1.289	1.658	1.980	2.358	2.617	2.860	3.160	3.373
∞	.253	.674	1.282	1.645	1.960	2.326	2.576	2.807	3.090	3.291

Source E. S. Pearson and H. O. Hartley, *Biometrika Tables for Statisticians*, Vol. 1. London: Cambridge University Press, 1966. Partly derived from Table III of Fisher and Yates, *Statistical Tables for Biological, Agricultural and Medical Research* published by Longman Group Ltd., London (previously published by Oliver & Boyd, Edinburgh, 1963). Reproduced with permission of the authors and publishers.

Appendix B

RECOMMENDED METHOD FOR AGGREGATE EVALUATION

A. Obtain representative sample.

   1. homogeneous crushed stone = 2,000 grams
   2. gravel = 6,000 grams

B. Wash sample throughly.

C. Examine general properties.

   1. overall condition
   2. average size and range of size
   3. angularity

D. Separate sample according to lithologic and textural variations.

E. Measure absorption.

F. Determine pore size distribution and Expected Durability Factor.

   1. Run only on classes with an absorption of greater than 1%.
   2. Verify normality of EDF data.

G. Compute minimum n required.

1. Recommend a 95% confidence coefficient and ± 5 EDF confidence interval, however other conditions can be used.
2. Follow method outlined in pages 42-44.
3. Calculate minimum n after four runs.
4. Make more porosimeter runs.
    a. If minimum n is sufficiently small make recommended number of runs.
    b. If minimum n is inconventiently large do one of the following:
        i) Subdivide sample more throughly.
        ii) Make more runs and recompute statistics.
5. Repeat procedure for other classes.

H. Compute total percent nondurable aggregate in source.

1. Add weight percents of all fractions with an average EDF of less than 50.
2. Use results to perdict durability.
    a. One criteria is Lindgren´s method.

Implementation Report

EVALUATION OF AGGREGATE SAMPLE DURABILITY

by

M. Hanson

The following suggestions are made as possible ways the results of this report might be implemented.

The main aim of this research was to determine the relationship between the required accuracy of a determination of the expected durability factor (EDF) of an aggregate sample, the degree of confidence that can be placed in the value obtained, and the number of mercury porosimetry runs required.

Appendix B summarizes the findings in the form of a suggested procedure to be used with an aggregate of unknown durability. The suggested parameters are a range of 5 in EDF at a confidence level of 0.95, but other choices may be appropriate for some instances.

It is suggested that this procedure be followed by IDOH on samples, first, with a well developed history of pavement performance. This would amount, essentially, to checking the researcher´s results, and is recommended because of the necessarily limited number of samples on which the results are so far based.

Probably the lithologic subdivision of a sample should be made, at least at first, by someone professionally trained in geology, although such a requirement is not a necessary aspect of the method. It also might be best to do initial tests on crushed stone samples, rather than on the more complicated gravels.

If the results obtained conform to the field history of the materials used, then the method should be expanded to new aggregate sources, with a possible view toward incorporating this procedure in specifications for aggregates.

COVER DESIGN BY ALDO GIORGINI